

08CL-5989
SP1-0035-C

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

O I P E S C
OCT 29 2004
PATENT & TRADEMARK OFFICE
Applicant: Eric Thomas Gohr et al.

Serial No.: 09/740,074

Filed: July 27, 2004

For: METHOD FOR REDUCING HAZE IN A
FIRE RESISTANT POLYCARBONATE
COMPOSITION

)
Group Art Unit: 1714
)
)
)
Examiner:
Szekely, Peter A.

DECLARATION PURSUANT TO 37 C.F.R. § 1.132

Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

I, Rajendra K. Singh, declare and state:

1. My educational background includes a B.S. in Chemistry from Bombay University, India, Masters of Science in Organic Polymer Chemistry from Virginia Commonwealth University and a Ph.D. in Chemistry from the University of Missouri-Rolla in Organic Polymer Chemistry.

2. I have been employed by the General Electric Company since October 1998, where I am currently a Product Engineer in the High Performance Polymers Technology Group at General Electric Plastics in Mount Vernon, Indiana.

3. I am an inventor or co-inventor on at least 5 U.S. patents assigned to the General Electric Company relating to plastic compositions, methods, and articles.

4. I am an inventor of the invention claimed in the above-identified application.

5. I am also an inventor on the parent application having U.S. Application Serial No. 09/749,645 filed on December 27, 2000, now U.S. Patent No. 6,730,720, which is hereby incorporated by reference in its entirety.

6. I designed the experiments in the two declarations – Declaration A and Declaration B that are attached to this document. These declarations were submitted in support of our invention during the prosecution of U.S. Application Serial No. 09/749,645 filed on December 27, 2000, now U.S. Patent No. 6,730,720. The experiments listed in the respective declarations were conducted using a polycarbonate, a potassium salt of perfluorobutane sulfonate (KFPBS) and a cyclic siloxane to form a fire retardant composition.

7. The experiments in Declaration A demonstrate that a) impact properties of polycarbonate resin are not degraded or changed in any manner by the addition of the potassium salt of perfluorobutane sulfonate b) the addition of the cyclic siloxane does not improve the impact properties of the composition. This clearly shows that the addition of the flame retardant salt in masterbatch form does not improve the impact properties as claimed by the Examiner.

7. The experiments conducted in Declaration B show that the melt volume rate increases with the increase in the cyclic siloxane content indicating a reduction in viscosity. The HDT test and the Vicat test also show a reduction in the softening temperature with increasing siloxane content. Such a reduction in softening temperature is detrimental to a flame retardant composition since it is generally desirable to have deformation occur at as high a temperature possible when heated or subjected to a flame. Thus from the above data, it is quite clear that the addition of the cyclic siloxane to the flame retardant polycarbonate composition containing a flame retardant salt does not improve the impact properties, but instead causes a decrease in the softening temperature of the composition which is generally undesirable.

8. The flame retardant salt used in the Declarations A and B is the potassium salt of perfluorobutane sulfonate, which belongs to the same family of salts as those claimed in Claim 1 and Claim 21. It is therefore expected that the salts claimed in Claim 1 and Claim 21 will behave in a manner similar to potassium salt of perfluorobutane sulfonate.

9. I further declare that all statements and representations made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements and representations were made with the knowledge that willful false statements and the like, so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued therefrom.

OCT 25 2004

Dated

Rajendra Kashiwath Singh

Rajendra K Singh, Ph.D.

8CL5989



DECLARATION A

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Eric Thomas Gohr et al.)
Serial No.: 09/749,645) Group Art Unit: 1714
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COMPOSITION) Examiner:
) Szekely, Peter A.

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2. I have been employed by the General Electric Company since October 1998, where I am currently a Product Engineer in the Lexan Technology Group at General Electric Plastics in Mount Vernon, Indiana.
3. I am an inventor or co-inventor on at least 2 U.S. patents assigned to the General Electric Company relating to plastic compositions, methods, and articles.

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4. I am an inventor of the invention claimed in the above-identified application.

5. I designed and supervised the preparation and testing of six compositions, which are shown in Table 1 and four additional compositions shown in Table 2. The compositions shown in Table 1 comprise polycarbonate resin, a flame retardant salt i.e., potassium salt of perfluorobutane sulfonate (KPFBS) and a cyclic siloxane as shown in Table 1. The compositions shown in Table 2 contain only polycarbonate resin and the flame retardant KPFBS. All the additives to the polycarbonate resin in Tables 1 and 2 are measured in parts per hundred (phr).

6. Five samples of each composition were prepared and tested according to the procedures described in the above-identified application. Samples were tested for Notched Izod impact strength as per ASTM D 256. The results of the tests have been averaged and are reported in Table 1 and Table 2.

Table 1

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
KPFBS (phr)	0.05	0.05	0.1	0.1	0.05	—
Siloxane (phr)	0.1	0.2	—	0.2	0.1	0.1
Notched Izod (ft-lb/inch)	15.694	15.873	15.427	15.492	15.578	15.419
Standard Deviation	1.141	0.646	0.705	1.301	0.558	0.992

7. As can be seen in Table 1, there is no observable trend in the impact strength, with either the mere presence or an increase in the amount of cyclic siloxane. For example, Samples 1, 2 and 5, all possess 0.05 phr of the flame retardant salt, while

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Samples 1 and 5 possess cyclic siloxane in an amount of 0.1 phr, and Sample 2 possesses the cyclic siloxane in an amount of 0.2 phr respectively. The impact strength increases only slightly from approximately 15.69 and 15.57 ft-lbs/inch for Samples 1 and 5 respectively to 15.87 ft-lbs/inch for Sample 2, which is within the limits of statistical variation when the standard deviations shown in Table 1 is taken into account. Similarly, Sample 3, which contains only 0.1 phr of the flame retardant salt may be compared with Sample 4 (which contains 0.1 phr of the flame retardant salt and 0.2 phr of the cyclic siloxane) and Sample 6, which contains 0.1 phr of the cyclic siloxane. From the impact strength results it can be seen that there is practically no variation in the impact strength results for Samples 3, 4 or 6. All the impact strength results are within the limits of statistical variation. This clearly shows that the addition of the flame retardant salt does not degrade impact strength in as much as the cyclic siloxane does not improve impact strength as claimed by the Examiner.

Table 2

	Sample 7	Sample 8	Sample 9	Sample 10	Sample 11
KPFBS (phr)	0.0	0.05	0.08	0.09	0.1
Notched Izod (ft-lb/inch)	15.060	15.093	13.674	15.48	16.13
Standard Deviation	0.441	0.677	0.629	1.027	0.976

8. The experiments shown in Table 2, were further conducted to prove that the addition of only the flame retardant salt to the polycarbonate resin does not in any way alter the impact properties of the polycarbonate resin. From the table it may be seen that increasing the quantity of the flame retardant salt from 0.0 to 0.1 phr does not reduce

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the impact properties of the polycarbonate resin. Sample 7, which does not contain any flame retardant salt has an impact strength of 15.060 ft-lbs/inch. Sample 9 does show slightly lower results, but this may be considered an outlier, since all the other samples have impact strength above 15 ft-lbs/inch. Thus, once again it can be clearly seen that the addition of the flame retardant salt to the polycarbonate resin does not degrade the impact strength and therefore the cyclic siloxane does not have to be added to improve the impact properties of the polycarbonate resin as maintained by the Examiner.

9. I further declare that all statements and representations made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements and representations were made with the knowledge that willful false statements and the like, so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued therefrom.

Dec 31'02

Dated

Rajendra K Singh

Rajendra K Singh, Ph.D.

DECLARATION B

8CL5989
(GP1-0035)



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Eric Thomas Gohr et al.)
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3. I am an inventor or co-inventor on at least 2 U.S. patents assigned to the General Electric Company relating to plastic compositions, methods, and articles.

DECLARATION B

8CL5989
(GP1-0035)

4. I am an inventor of the invention claimed in the above-identified application.

5. I designed and supervised the preparation and testing of six compositions, which are shown in Table 1. The compositions comprise polycarbonate resin, 0.08 parts per hundred (phr) of the flame retardant salt potassium salt of perflouorobutane sulfonate (KPFBS) and a variable amount of cyclic siloxane from 0.05 to 2 phr based on total weight of the composition as shown in Table 1.

6. Five samples of each composition were prepared and tested according to the procedures described in the above-identified application. Samples were subjected to tensile testing as per ASTM D 638-99 to determine the modulus, maximum stress at yield and break, and elongation at yield and break. Samples were tested for Notched Izod impact strength as per ASTM D 256. Flexural Modulus and flexural strength were measured as per ASTM D 790. Heat distortion temperature (HDT) was measured as per ASTM D 648. The results of the tests have been averaged and are reported in Table 1.

7. As can be seen in Table 1, almost all mechanical properties such as the flexural modulus, secant modulus, flexural strength, flex stress at 5% strain, impact strength, ductility, impact energy, ratio of energy to maximum load, and tensile properties show virtually no change with the increase in the amount of cyclic siloxane. The average values for all of these mechanical properties are almost identical within the limits of statistical variation, irrespective of the amount of cyclic siloxane. Further there is no observable trend in the mechanical properties, especially in the impact strength, with the increase in the amount of cyclic siloxane.

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8. The only properties that show any trends that are dependent upon the amount of cyclic siloxane are the melt volume rate at 18 minutes and 6 minutes as well as the heat distortion temperature (HDT) test and the softening temperature derived from the Vicat test (ASTM D 1525). While the melt volume rate increases with the increase in cyclic siloxane content indicating a reduction in viscosity, it can clearly be seen that the HDT test and the Vicat test show a reduction in the softening temperature with increasing siloxane content. Such a reduction in softening temperature is detrimental to a flame retardant composition since it is generally desirable to have deformation occur at as high a temperature possible when heated or subjected to a flame. Thus from the above data, it is quite clear that the addition of the cyclic siloxane to the flame retardant polycarbonate composition containing a flame retardant salt does not improve the impact properties, but instead causes a decrease in the softening temperature of the composition which is generally undesirable.

9. I further declare that all statements and representations made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements and representations were made with the knowledge that willful false statements and the like, so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued therefrom.

OCT 24 2002

Dated

Rajendra Kasturbhai Singh
Rajendra K Singh, Ph.D.

DECLARATION B

TABLE 1

Sample No.	1	2	3	4	5	6
FR Salt Loading (phr)	0.08	0.08	0.08	0.08	0.08	0.08
Cyclic Siloxane Loading (phr)	0.05	0.1	0.25	0.5	1	2
Properties	Units	1	2	3	4	5
Melt Volume Rate/ 18 min.	cm ³ /10 min	11.68	13.17	13.19	14.59	15.26
Melt Volume Rate/ 6 min.	cm ³ /10 min	11.32	12.68	12.76	13.8	14.88
Flexural Modulus	PSI	337000	343000	339000	344000	343000
Secant Modulus@22%Strain	PSI	325000	327000	324000	329000	327000
Flexural Strength	PSI	14600	14500	14500	14600	14600
Flex Stress@5%Strain	PSI	13100	13000	13000	13100	13000
HDT Temperature	°C	127.4	126	126.9	125.2	124.7
Ductility	%	100	100	100	100	100
Impact Strength	lbf/in	16.463	16.004	16.463	15.745	15.837
Ductility	%	100	100	100	100	100
Drop Velocity	ft/s	11.72	11.72	11.72	11.73	11.72
Impact Energy	ft-lbf	106.8	106.8	106.8	106.9	106.8
Energy to Max Load	ft-lbf	52.2	52.4	52.5	53.3	54
Total Energy	ft-lbf	56.2	56.6	56.5	57.2	58
Max Load	lbf	1580	1610	1610	1610	1610
Tensile Modulus	PSI	336000	339000	334000	337000	340000
Tensile Stress@Yield	PSI	8920	8900	8920	8900	8910
Tensile Stress@Break	PSI	9920	10300	10100	9660	10000
Tensile Elongation@Yield	%	6.36	6.2	6.28	6.3	6.22
Tensile Elongation@Break	%	126.42	134.52	142.1	144.18	147.22
Cdn: Specimen Thickness (125 mils)	p[FTP]	0.9935	0.9558	0.967	0.9406	0.9036
Vicat Temperature	°C	142.9	142.5	142.8	141.9	141.5
Yellowness Index	-	2	1.5	1.5	1.4	1.6
Transmission	%	90.2	90.7	90.8	90.8	90.5
Haze	%	1.5	1	0.8	0.8	1.2